

DEVELOPMENT OF A NICKEL BASE ALLOY SHEET  
FOR HIGH TEMPERATURE APPLICATION



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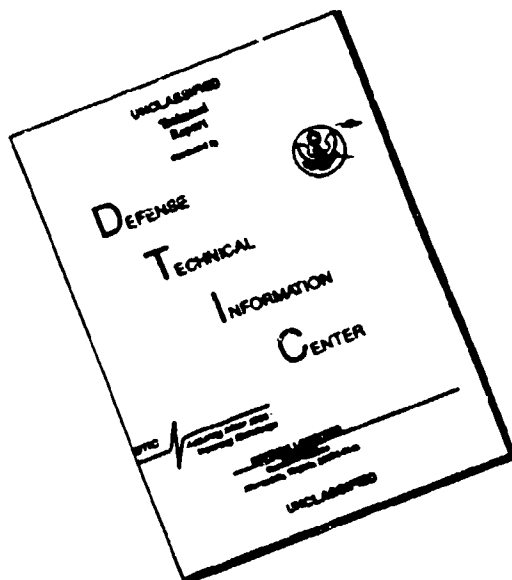
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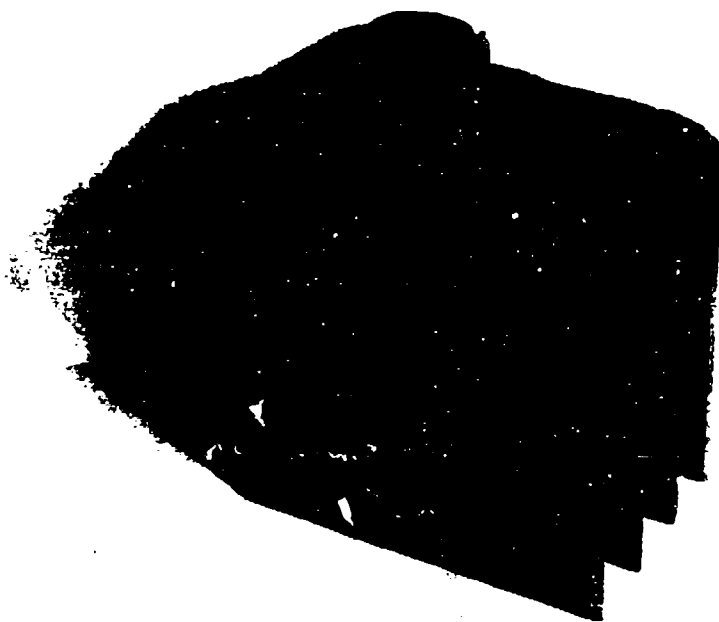


Figure 1. A Typical Inco 713c Sheet Blank Casting.

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across the sheet was greatly reduced. With this improved uniformity of load application, hard low ductility materials which were impossible to roll in conventional mills could be rolled. The basic rolling procedure was as follows:

1. The cast sheet blank, normally 4 inches by 6 inches by .125 inches was cleaned by vapor blasting and any casting flash was removed by hand grinding. Cast sheet blanks over 4 inches wide required surface grinding to remove thickness variations.

2. The cleaned cast sheet blank was then heated to 1950°F in air. To minimize oxidation the sheet blank was left in the furnace only long enough for it to come to temperature.

3. The sheet was then rolled in the mill at reductions of 1 to 3 mils per pass with reheating to 1950°F between passes.

4. After rolling the oxide scale formed by the repeated furnace cycles was removed by sand blasting.

5. Each sheet was then X-rayed to determine the extent and location of internal defects. A representative photograph of rolled sheet is shown in Figure 2.

#### C. HEAT TREATMENT

The heat treatment used on the Inco 713c specimens in Tables 1 and 2 consisted of a 2200°F solution anneal for 24 hours under an argon atmosphere with air cooling. The Rene' 41 specimens in Tables 3 and 4 were solution treated in air at 1950°F for 15 minutes, air cooled and then aged at 1400°F for 16 hours. Heat treatments used on the Inco 713c specimens in Table 5 consisted of a solution treatment at 2200°F for 4 hours in argon, air cooling and aged at 1450°F for 16 hours. A portion of the specimens received a subsequent solution treatment at 2200°F for 2 hours.

#### D. TESTING

The mechanical test portion of this program consisted of tensile and stress-rupture tests. All of the mechanical tests in this report were performed by Metcut Research Associates, Inc. of Cincinnati, Ohio. Inco 713c and Rene' 41 specimen blanks 2 inches by 8 inches by .040 inches in the heat treated condition were prepared by LTV and shipped to Metcut. The Inco 713c specimens were cut selectively from areas on the rolled sheet which showed no evidence of cracking or other discontinuities. Tensile and stress-rupture specimens of the configuration shown in Figure 3 were fabricated by Metcut from these blanks.

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TABLE 1

## TENSILE TESTS ON INCO 713c SHEET

Specimen No.	Temperature °F	Heat No.	U.T.S. ksi	0.2% Y.S. ksi	Elongation %
C-1	1700	594	91.3	67.8	2.0
C-2	1700	594	70.5	68.0	1.0
C-3	1700	594	61.6 (a)	--	0.5
C-4	1700	594	87.4	69.7	1.0
C-5	1700	594	93.0	65.3	1.5
C-6	1900	594	50.7	31.1	5.0
C-7	1900	594	48.0	30.5	4.0
C-8	1900	594	51.1	31.4	5.0 (b)
C-9	1900	594	55.0	36.1	3.0
C-10	1900	594	51.3	33.6	3.0

Heat Treatment: 2000°F for 24 hours in an argon atmosphere, air cool

Strain rate through 0.2% Yield: 0.005 in/in/min

Head rate thence to failure: 0.05 in/in/min

- (a) Broke before reaching 0.2% yield strength in radius outside gage section
- (b) Broke outside gage section

TABLE 2

## STRESS-RUPTURE TESTS ON INCO 713c SHEET

Specimen No.	Temperature (°F)	Heat No.	Stress (ksi)	Rupture Life (hours)	Elongation (%)
C-45	1700	598	30.0	F.O.L. (a)	1.1
C-40	1700	598	25.0	16.1	0.8
C-41	1700	598	25.0	20.6	0.8
C-42	1700	598	25.0	46.8	1.6
C-43	1700	598	25.0	2.7	nil
C-11	1700	594	25.0	33.5	4.0
C-46	1700	598	23.5	46.6	1.2
C-47	1700	598	23.5	5.1	nil
C-48	1700	598	23.5	58.7	2.0
C-31	1700	594	23.5	33.7	4.2
C-32	1700	594	23.5	44.0	3.9
C-49	1700	598	22.0	89.6 (b)	3.9
C-50	1700	598	22.0	74.7	1.6
C-51	1700	598	22.0	63.3	2.7
C-52	1700	598	22.0	28.3	0.8
C-53	1700	598	22.0	45.0	0.8
C-18	1800	595	13.1	59.9	4.4
C-19	1800	595	13.0	91.0	5.3
C-20	1800	595	13.0	73.5	4.6
C-21	1800	595	13.0	63.6	3.3
C-22	1800	597	13.0	52.5	0.4
C-28	1800	597	13.0	15.6	0.8
C-33	1800	598	14.0	13.1	0.8
C-34	1800	598	14.0	F.O.L. (a)	0.3
C-35	1800	598	14.0	31.7	1.1
C-36	1800	598	14.0	56.0	3.4
C-37	1800	598	14.0	75.4	4.2
C-38	1800	598	14.0	92.0	4.8
C-23	1800	597	15.0	22.9	1.5
C-24	1800	597	15.0	36.0	2.3
C-25	1800	597	15.0	35.3	0.7
C-26	1800	597	15.0	17.1	0.7
C-27	1800	597	15.0	37.6	1.8
C-13	1900	595	17.0	(c)	3.1
C-14	1900	595	15.0	6.0	4.2
C-15	1900	595	10.0	24.8	3.1
C-16	1900	595	7.0	56.1	13.3
C-17	1900	595	4.0	503.7	11.4

Heat Treatment: 2000°F for 24 hours in argon atmosphere,  
air cool

- (a) Failed on Loading
- (b) Temperature 35°F low for first 20 hrs.
- (c) Timer did not shut off. Rupture life at least 1.6 hours but not more than 17.4 hours.



TABLE 3

## TENSILE TESTS ON RENE' 41 SHEET

Specimen No.	Temperature °F	U.T.S. ksi	0.2% Y.S. ksi	Elongation %
R-1	1700	60.8	43.5	22.0
R-2	1700	58.0	42.9	18.0
R-3	1700	59.4	45.6	18.5
R-4	1900	15.6	9.50	36.5
R-5	1900	15.9	9.73	36.0
R-6	1900	16.2	9.99	41.0

Heat Treatment: Solution at 1950°F for 15 minutes, air cool  
Age at 1400°F for 16 hours

Strain rate through 0.2% Yield: 0.005 in/in/min--

Head rate thence to failure: 0.05 in/in/min

TABLE 4

## STRESS-RUPTURE TESTS ON RENE' 41 SHEET

Specimen No.	Temperature °F	Stress ksi	Rupture Life (hrs)	Elongation %
R-13	1700	16.0	28.8	18.5
R-14	1700	16.0	28.3	17.8
R-17	1700	13.5	61.2	20.8
R-18	1700	13.5	63.2	19.1
R-19	1700	11.0	119.4	16.4
R-20	1700	11.0	141.3	14.5
R-9	1800	12.5	7.7	17.7
R-8	1800	9.5	21.4	18.7
R-7	1800	9.5	25.0	20.6
R-10	1800	8.0	42.2	18.5
R-11	1800	8.0	35.1	23.4
R-12	1800	6.5	97.3	18.9
R-21	1800	6.5	91.1	23.2

Heat Treatment: Solution at 1950°F for 15 minutes, air cool  
Age at 1400°F for 16 hours

TABLE 5

## TENSILE TESTS ON RECRYSTALIZED INCO 713c SHEET

Spec. No.	Temp. °F	Heat No.	Heat-Treat	U.T.S. ksi	0.2% Y.S. ksi	Elongation %
C-106	1700	600	1	67.0	62.5	1.0
C-107	1700	600	1	93.5	61.5	3.0
C-112	1700	599	1	89.5	65.4	2.0
C-113	1700	600	1	89.5	62.4	1.5
C-108	1700	600	2	97.2	73.6	1.5
C-114	1700	600	2	98.9	70.4	5.0
C-115	1700	600	2	95.4	68.2	5.0
C-116	1700	600	2	91.5	67.2	3.0
C-101	1900	599	1	46.2	32.0	3.0
C-102	1900	599	1	51.8	34.7	2.7 (a)
C-103	1900	599	1	51.0	31.3	3.9 (a)
C-104	1900	599	1	48.8	36.0	2.0
C-105	1900	599	2	45.5	28.7	4.7 (a)
C-109	1900	599	2	45.5	27.6	9.5
C-110	1900	599	2	48.4	29.4	6.5
C-111	1900	599	2	45.8	27.8	8.5

(a) Broke in reduced section but outside of gage length

## Heat Treatments:

1. 2200°F for 4 hours in argon, air cool  
1450°F for 16 hours in air, air cool
2. 2200°F for 4 hours in argon, air cool  
1450°F for 16 hours in air, air cool  
2200°F for 2 hours in argon, air cool

Strain rate through 0.2% yield: 0.005 in/in/min  
Head rate thence to failure: 0.05 in/in/min



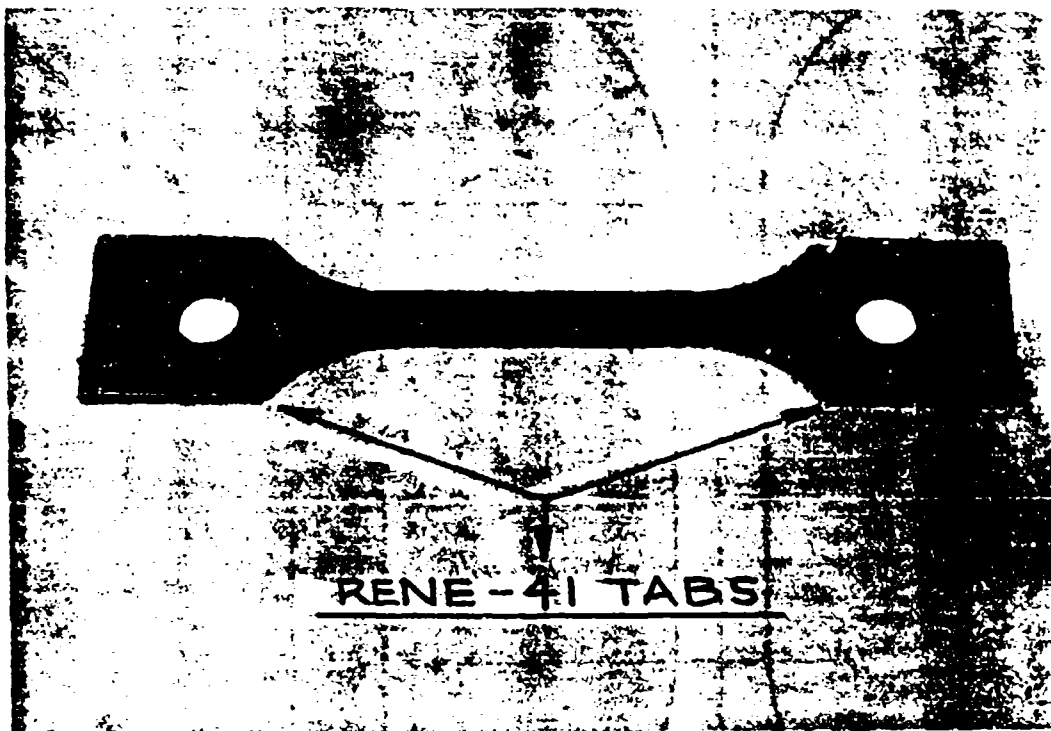


Figure 4. A Representative Stress-Rupture Specimen  
Showing the Reinforcing Tabs.

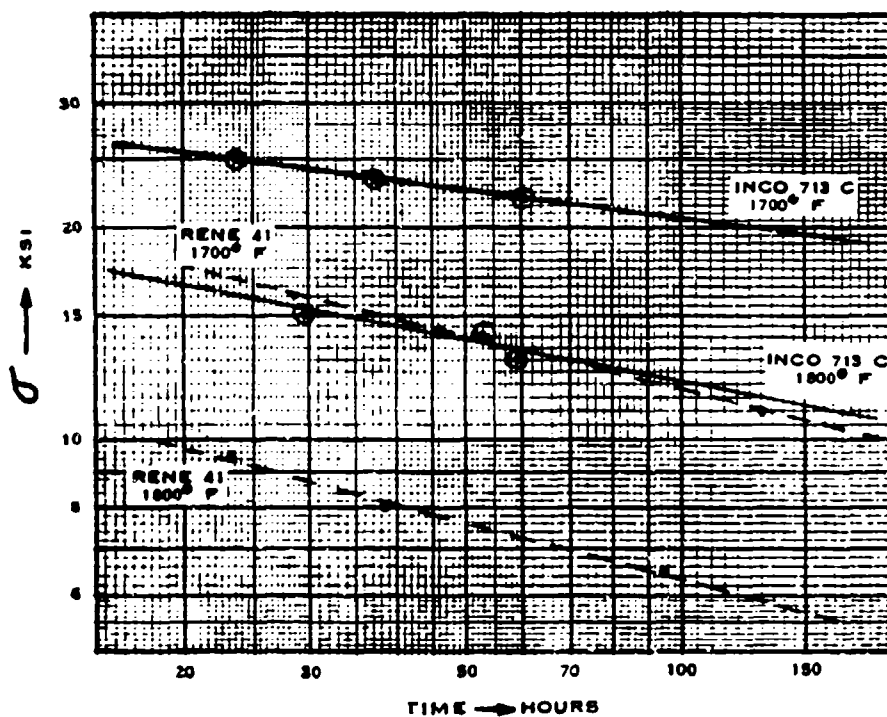


Figure 5. Stress-Rupture Test Results

TABLE 6

## CHEMICAL ANALYSES OF FOUR HEATS OF INCO 713c SHEET

<u>Element</u>	<u>Heat 594</u>	<u>Heat 595</u>	<u>Heat 597</u>	<u>Heat 598</u>
Nickel plus Cobalt	70.67	70.59	70.66	71.21
Chromium	13.45	13.43	13.49	13.54
Molybdenum	5.38	5.38	5.40	5.38
Aluminum	6.26	6.23	6.32	6.27
Columbium plus Tantalum	2.29	2.31	2.27	2.28
Titanium	0.84	0.81	0.83	0.84
Carbon	0.14	0.14	0.12	0.14
Silicon	0.10	0.10	0.12	0.11

F.

#### METALLURGICAL EVALUATIONS

Metallographic specimens representative of all sheet conditions were prepared and examined to evaluate the microstructure of the alloy as a function of fabrication history. All fracture surfaces were examined optically at low power (1X to 7X). Representative cross-sections of fracture surfaces were examined metallographically to evaluate microstructural differences between specimens with high and low mechanical properties. In addition representative specimens were examined at high magnification using an electron microscope. The etchant found most effective for Inco 713c specimens was Marbles reagent of the following composition:

$\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$	12 gm
HCl	60 cc
$\text{H}_2\text{O}$	60 cc

#### IV. DISCUSSION

A.

##### ROLLING

1. It was determined early in the program that it would be impractical to roll 8 inch wide sheet. The basic reason being that a sheet of this width required more power to roll than the mill was capable of delivering. As a result the sheet would break up badly and could not be effectively reduced to the .040 inch thickness. Therefore, the sheet width was reduced to 6 inches which was a rollable width. The 6 inch sheet width was more satisfactory than the 8 inch width, however, most of the sheets would develop cracks or other surface defects to some degree. Although several specimens could be cut from each sheet, they could not be cut indiscriminately from any part of the sheet. It was necessary to select the location of the specimens such that there were no cracks or defects near the gage section. Approximately 50% of the specimens used in this program were cut from a 6 inch wide sheet. The remaining specimens were obtained from 4 inch wide sheet. There were no apparent differences in the physical properties of specimens from the different sheet widths. The differences in rolling the 6 inch wide sheet and the 4 inch wide sheet were basically problems of first obtaining perfectly sound sheet and, second, of requiring a considerably longer time to prepare and roll the 6 inch wide sheet.

2. The 4 inch wide sheet was the optimum width for the rolling mill equipment available to this program. The 4 inch wide sheet could be rolled in a reasonably short time with

reproducibly crack free sheets being produced. Approximately half as many rolling passes were required to reduce a 4 inch wide sheet from .125 inches to .040 inches as were required to reduce a 6 inch wide sheet the same amount. Less time was required for preheating narrower sheet due to its smaller mass and a higher specimen yield per cast blank was obtained. That is, each cast sheet blank yielded only one 6 inch wide sheet from which a maximum of 6 specimens could be obtained; however, the same cast sheet blank would yield two 4 inch wide sheets from which a maximum of 8 specimens could be obtained. In addition, the cracks in the 6 inch wide sheets generally reduced the specimen yield per sheet, making the 4 inch wide sheet distinctly more efficient.

Several significant comments should be made concerning various aspects of the rolling of Inco 713c in this program:

a. Thickness Variations - Even though the design of the LTV rigid rolling mill eliminated the major contribution to crowning in the rolled sheet; that is, it eliminated bearing deflection; a certain amount of crowning did result from deflection of the rolls themselves. This caused a tendency to elongate the edges of the cast sheet blank more than the center of the sheet. As a result, very large tensile stresses were retained in the center part of the sheet during the early stages of rolling. In the wider sheets this effect was more pronounced and was sufficient to cause cracks in the middle portion of the sheet. The cracks were intergranular due to the low grain boundary ductility of the cast structure in the undeformed portion of the sheet. Thickness variations of greater than .005 inches across the sheet also resulted in cracks for the same reason. If a thickness variation occurred, the thicker material would be reduced and elongated while the thinner sections would remain in the cast condition. Tensile stresses exerted by the elongated material on the cast structure were sufficient to cause cracks in all cases when the thickness variations were greater than .005 inches and in some cases when the variation was less.

The thickness variations were a serious problem in rolling of the 6 inch wide sheet and required that the sheet be surface ground prior to rolling to remove the thickness variations. The 4 inch wide sheet, however, had a greater tolerance for thickness variations and did not require surface grinding. Even in the few cases in which .005 inch thickness variations did occur, the crowning effect was sufficiently small that cracking did not occur. The 4 inch wide sheets were not surface ground other than the small amount of hand grinding required to remove casting flash, but were only vapor blasted as a preparation for rolling.



Grinding of 6 inch wide sheets was complicated by the difficulty experienced in attaching the .125 inch cast sheet to the grinding table. The most satisfactory method was found to be the use of doublebacked adhesive tape, however, this technique required great care and even then was unreliable. Several glues and other adhesives were evaluated but were uniformly unsuccessful as reliable methods of attaching the sheet to the grinding table. It was found to be advisable to entirely avoid the grinding operation if at all possible. By eliminating the grinding requirement the change from the 6 inch to the 4 inch wide sheet greatly reduced the time and effort required to produce a rolled sheet.

b. Edge Cracks - Small edge cracks were observed to occur early in the rolling process but they did not tend to propagate themselves beyond approximately 1/8 of an inch. In cases where thickness variations occurred at the edge of the cast sheet, cracks were occasionally found to extend as far as 1/2 inch into the sheet. However, this was the exceptional case. Edge cracks were not trimmed during rolling but were allowed to remain on the sheet until after the final rolling pass.

c. Grain Growth - In rolling the 6 inch wide sheet it was necessary to expose the sheet to the 1950°F heating cycle many more times than was necessary with the narrower sheet. Since the reductions per pass were limited to .001 to .003 inch there was not sufficient reduction per pass to promote recrystallization of the sheet as in the case of conventional hot rolling operations. The combination of the low reduction per pass and the absence of recrystallization permitted many of the as-cast grains to persist and possibly grow somewhat during the rolling operation. As a result a very large grain size resulted in the final hot rolled sheet. This presented a serious problem since the grain boundaries were the preferred failure location in this material. Large grain sizes then created grain boundaries which in some cases extended entirely across the cross-sectional area of the test specimen gauge section. Typical sizes of these large grains were .25 inch wide by 1 inch long after hot reduction of approximately 70%. Even though the 4 inch wide sheet could be rolled somewhat more rapidly with greater work applied to the structure per pass, the rolling mill still did not have sufficient power to impart the necessary degree of deformation per pass to the sheet to cause recrystallization during each rolling cycle. In some cases evidence of local recrystallization was observed; however, many very large grains were retained in the final sheet. It was therefore concluded that the reduction per pass was insufficient to reliably cause recrystallization during the hot rolling process.

It was observed that in order to avoid cracking of the cast sheet blank it was necessary during the early stages of rolling to limit the reduction per pass to a maximum of .002 inch or approximately 1% of the sheet thickness. However, after a reduction of approximately 30% the sheet became much more tolerant to the rolling process and considerably greater reductions per pass were then possible, in fact the permissible reduction per pass from that point on exceeded the capacity of the rolling mill. The mill would stall if reductions greater than approximately 5% per pass were attempted. It is probable that a more powerful rolling mill would be capable of considerably greater reductions per pass without cracking the Inco 713c sheet. All cracking observed during this program occurred during the early stages of rolling. The formation of new cracks was not observed after the first 10% reduction. It was felt that if reductions per pass of the order of 10% could be obtained then a closer approach to true hot rolling, in which recrystallization occurs during each rolling pass, would be accomplished.

#### B. HEAT TREATMENT

Reference 1 included the use of several prospective heat treatments for this material, however, there was insufficient test data at any given condition to accurately ascertain the effects of the various heat treatments on the mechanical properties. However it appeared that the best properties were obtained after a solution anneal at 2200°F for 24 hours. Therefore this heat treatment was used on all Inco 713c specimens in Tables 1 and 2.

From observations of microstructures it appeared that some local recrystallization occurred as a result of this treatment, however, in other areas grains appeared to have remained in their original as-cast size or possibly even grown slightly. This was reasonable since it would be expected that only a small amount of internal energy would be retained in the sheet after the hot rolling operation. This small amount of energy would represent a marginal condition for recrystallization thereby permitting a few recrystallized grains to form. In other grains the strain energy would be relaxed by the formation of a polygonized sub-grain structure within the as-cast grain or by a small amount of grain growth. A structure composed of both small grains and very large grains would thereby result. The 2200°F treatment probably did little more than dissolve and redistribute in a more uniform manner the various constituents of the alloy and relieve rolling stresses retained in the sheet. It did not cause significant recrystallization of the microstructure.

#### C. TEST RESULTS

##### 1. TENSILE TESTS

A comparison of Tables 1 and 3 clearly shows that

Inco 713c is a much higher strength alloy than Rene' 41, however the ductility of Inco 713c was below normal acceptable levels for sheet alloys. The average ultimate tensile strength of the Inco 713c at 1900°F (51,200 psi) was more than three times that of Rene' 41 under the same conditions. Since Rene' 41 was considered one of the strongest high temperature sheet alloys currently available, the results of these tests indicate that Inco 713c sheet represents a significant improvement in the elevated temperature strength of nickel base sheet alloys.

The test data in Reference 1 suggested ultimate strength values in the 50,000 psi range at 1900°F but test conditions were not controlled as closely as in the present program. The results reported in Table 1 were obtained from specimens in which the test temperature over the gauge length was held to within  $\pm 5^\circ\text{F}$ , all specimens were taken from the same heat and all specimens received the same heat treatment (2200°F for 24 hours under argon and air cool). Therefore considerable reliability can be placed on the results of this program. The close agreement of the values in Table 1 is a confirmation of this reliability. The cause of the low ductility observed in the 1700°F tensile tests could not be determined precisely within the limitations of this program. However, it would appear that in this temperature range the precipitation process involving the Ni-Ti-Al complex could cause an over-aged condition to develop in which the size and distribution of the precipitated particles, especially those associated with the grain boundaries, could cause a reduction in ductility. The 1900°F test temperature was probably sufficient to take this complex back into solution, hence improving the ductility of the alloy. The low tensile ductility at 1700°F corresponded to data reported in Reference 2. Although that data was taken from cast material and the observed elongation was at a higher level, the relatively low value at 1700°F does confirm the mutual relationship of the 1700° and 1900°F results reported in Table 1. That is, the observation of relatively low ductility in the neighborhood of 1700°F appears to be inherent property of the Inco 713c material and not a result of the fabrication process described in this report.

The significant observations regarding the tensile test results were:

a. Inco 713c sheet produced by the process described in this report demonstrated an average ultimate tensile strength in excess of 50,000 psi at 1900°F with reasonable reproducibility.

b. The results of this second group of tensile tests (Table 5) indicate that improvements in mechanical properties are possible by minor changes in the rolling and heat

treating procedures. Therefore, additional experimental work would be expected to result in even further ductility improvements.

## 2. STRESS-RUPTURE TESTS

The stress-rupture tests were initially intended to be conducted at 1700°F and 1900°F, however the initial tests at 1900°F indicated that the stress levels required for reasonable rupture times were too low to be of practical use. Hence the higher temperature was decreased from 1900° to 1800°F. The results of these tests are reported in Table 2. Rene' 41 specimens tested at the same temperatures required substantially lower stresses to obtain similar times to rupture (see Table 4) again demonstrating the higher strength of the Inco 713c sheet. It was apparent that the ductility of the Inco 713c specimens as measured by the elongation at fracture was low and unpredictable.

Metallographic work conducted in an effort to determine the cause of the low ductility led to the discovery that in each case where low stress-rupture ductility occurred, a single very large grain existed at the fracture surface. Figure 6 is a photomicrograph of a cross-section through the fracture surface of specimen C-47 which failed with low ductility and shows a very large grain at the fracture surface. Each of the other stress-rupture specimens which failed with low ductility had similar microstructures.

It was also apparent that the failure occurred preferentially in the grain boundaries of the unusually large grains. A visual examination of matching surfaces of fracture specimens showed a relatively smooth convex area on the side containing the large grain which differed slightly in color and texture from the surrounding area. In each case this visual observation corresponded to the actual presence of a large grain as confirmed by metallographic examination of cross-sections through fracture surfaces, such as the one shown in Figure 6. The opposite side of the fracture surface was relatively small grained in each case.

## 3. DUCTILITY IMPROVEMENT

Since the grain boundaries of large grains were preferred failure sites, it was apparent that the first step in improving the ductility of the alloys would be to refine the grain size. Refinement of the grain size by recrystallization required that sufficient internal strain energy (cold work) be imparted to the microstructure to permit the nucleation and growth of new grains. Observations of the microstructure at various points during hot-rolling showed no evidence of a strained structure. Apparently all strain energy was being

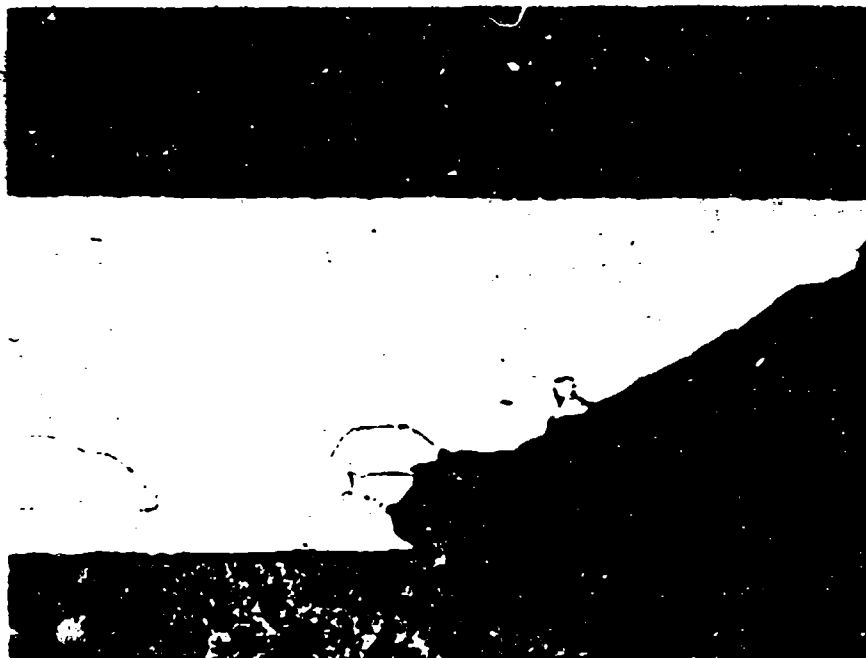
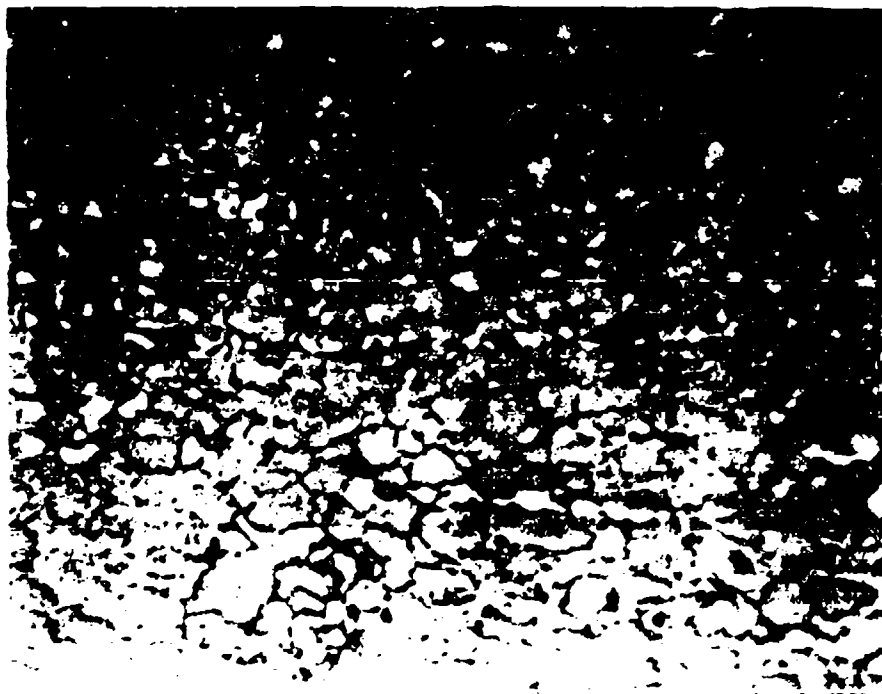


Figure 6. Photomicrograph of a Cross-Section Through the Fracture Surface of a Specimen (C-47) Which Failed With Low Ductility. 50X



CONDITION:

Hot-rolled 85%, cold-rolled 12.5%

Heat Treatment:

Solution: 2200°F, 4 hours in argon, air cooled

Age: 1500°F, 16 hours

Figure 7. Photomicrograph of Inco 713c Showing Recrystallized Structure. 1000X

annealed out by the heating cycles between rolling passes. It was apparent that recrystallization could not occur without imparting a greater amount of cold work to the structure than that remaining after the hot rolling process.

It was observed that an additional 12.5% cold reduction after the hot rolling procedure followed by heat treatment at 2200°F for 4 hours in argon was required to promote uniform recrystallization of the microstructure. Figure 7 shows the microstructure of a specimen which had been cold rolled 12.5%, solution heat treated at 2200°F for 4 hours and overaged at 1500°F for 16 hours. The purpose of the overaging treatment was to facilitate etching by sensitizing the structure. The solution treated structure could not react with the etchant sufficiently well to bring out the grain boundaries. A comparison of the grain sizes in Figure 6 and 7 clearly shows that at least an order of magnitude improvement in grain size was accomplished and all evidence of the exceptionally large grains was removed. Although the 12.5% cold reduction followed by 4 hours at 2200°F was sufficient to recrystallize the structure, some variation in recrystallized grain size was observed. However, it was clear in all cases that the recrystallized structure was a great improvement over the hot-rolled structure.

It was not possible within the scope of this program to determine the cold reduction-heat treatment combination which would develop the optimum grain size or the best mechanical properties in the rolled sheet.

The test results reported in Table 5 were obtained from sheet which had been hot rolled from a thickness of 0.125 inches to 0.040 inches, cold rolled to 0.035 inches (12.5% cold reduction), solution heat treated under argon at 2200°F for 4 hours, air cooled, and aged at 1450°F for 16 hours. Eight of the specimens had received an additional solution treatment at 2200°F for 2 hours to evaluate the difference between the aged and solution treated conditions. At each test temperature it was apparent that the solution treated specimens were more ductile than the aged specimens. The aged specimens were somewhat stronger at 1900°F than the solution treated specimens but the opposite was true at 1700°F. It was clear from these test results that significant improvements in ductility resulted from the cold rolled and recrystallized specimens in the solution treated condition, however the reported experimental data was limited in scope and should not be considered conclusive. It is highly probable that additional experimental work in the areas of rolling schedule and heat treatment will result in even greater improvements in strength and ductility.

Several specimens were examined by electron microscopy to observe the microstructure at high magnifications. It was difficult to control the etching characteristics of the alloy sufficiently well to obtain completely reproducible microstructures, however, several observations should be noted.

Three distinct phases were observed in the Inco 713c microstructure; a smooth rounded particle such as shown in Figure 8, a rectangular particle apparently aligned crystallographically as in Figures 9 and 10, and a background matrix structure. Although these particles have not been positively identified it appeared that the smooth rounded particles were probably carbides since they were present in all specimens and resembled carbide particles in other alloys. Their size and distribution was quite variable from specimen to specimen. The rectangular particles were assumed to be the  $\text{Ni}_3(\text{Al}, \text{Ti})$  complex since this type microstructure in other nickel base superalloys has been identified as  $\text{Ni}_3(\text{Al}, \text{Ti})$  and since the observed volume of these particles was approximately the same as the expected volume of  $\text{Ni}_3(\text{Al}, \text{Ti})$  in the alloy. These particles were not observed in all microstructures and time did not permit investigation of the conditions under which they occurred or did not occur.

Electron photomicrographs of each of the two heat treat conditions for which test results are presented in Table 5 are shown in Figures 9 and 11. The re-solutioned structure does not show the rectangular particles which indicates that they have been taken back into solid solution. This implies that the best high temperature ductility in this alloy is obtained when carbides are the only particles existing in the microstructure.

## V. RECOMMENDATIONS

Since the elevated temperature mechanical properties of Inco 713c represent a significant improvement over the corresponding properties of Rene' 41, the continued development of a suitable process for rolling Inco 713c sheet is recommended. This development program should include the following.

A. Additional effort should be directed toward determining the rolling schedule-heat treatment combination which results in optimum properties in the sheet material. This program should include investigation of the metallurgical factors affecting the mechanical properties.

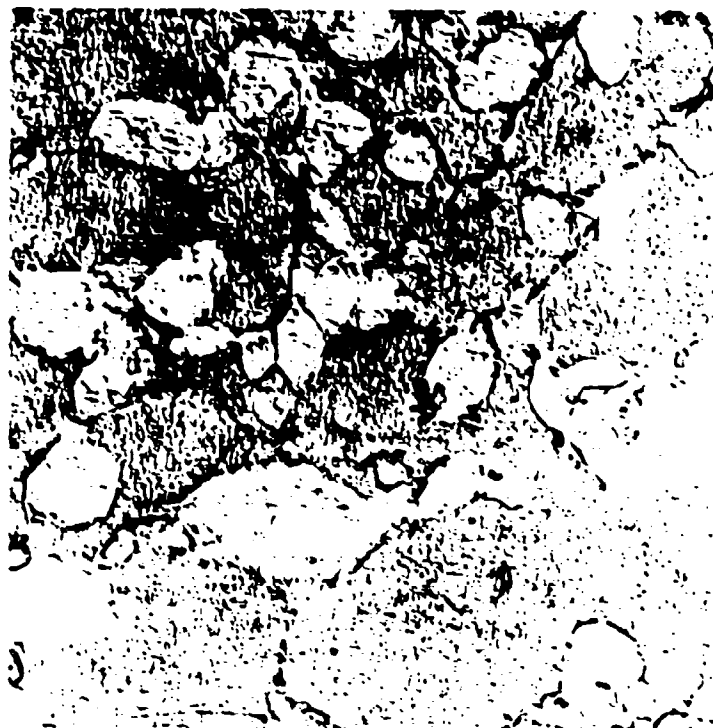


Figure 8. Photomicrograph of Same Specimen as Figure 7 at Higher Magnification. 6000X



CONDITION:

Hot-rolled 85%, cold-rolled 12.5%

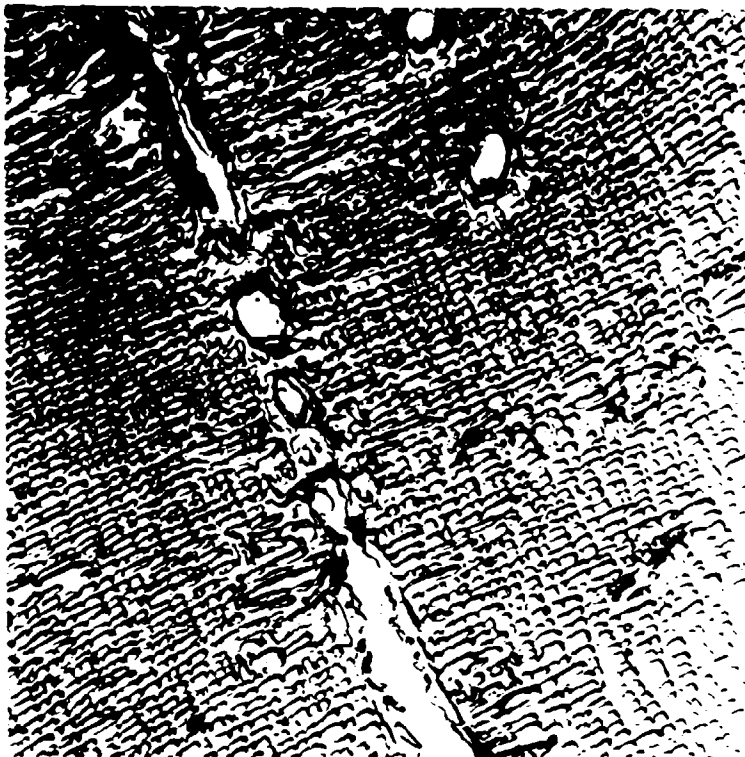
Heat Treatment

Solution:  $-2200^{\circ}\text{F}$ , 4 hours in argon, air cooled

Age:  $1450^{\circ}\text{F}$ , 16 hours

Figure 9. Photomicrograph of Inco 713c Showing Rectangular Particles. 6000X





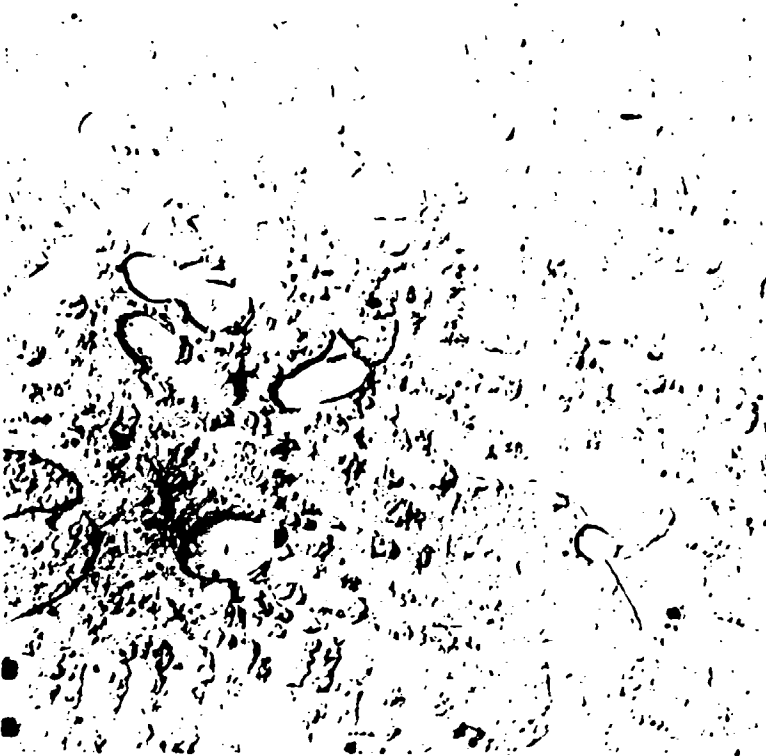
**CONDITION:**

Hot-rolled 85%, cold-rolled 25%

Heat Treatment:

Solution: 2200°F, 4 hours in argon,  
air cooled

Figure 10. Photomicrograph of Inco 713c Showing Carbides and Rectangular Particles. 6000X



**CONDITION:**

Hot-rolled 85%, cold-rolled 12.5%

Heat Treatment:

Solution: 2200°F, 4 hours in argon, air cooled

Age: 1450°F, 16 hours

Re-solution: 2200°F, 2 hours in argon,  
air cooled

Figure 11. Photomicrograph of Inco 713c After a Re-solution Treatment Showing Absence of Rectangular Particles. 6000X

6000X

B. A more detailed mechanical properties evaluation, including a wider range of test conditions, should be conducted to accurately determine the characteristics of Inco 713c produced by the optimum process.

C. Since the same basic techniques used in this program should be applicable to other high strength, difficult-to-roll alloys, a program should be conducted to determine whether or not other alloys in that category might be desirable in sheet form. An investigation of the feasibility of rolling these alloys using the basic process described in this report should then be conducted.

D. If the results of recommendations A and B indicate that strength properties greater than 50% above those of existing nickel base alloys are obtainable in the Inco 713c alloy sheet, then a program should be conducted toward scaling up the present process for the production of wider sheet. Such a program would involve the casting of larger sheet blanks and the use of more powerful rolling equipment. This program should also include the rolling of sheet thicknesses down to the foil gauges.

## VI. REFERENCES

1. ASD-TDR-62-869, "Development of a Nickel Base Alloy Sheet for High Temperature Application" by H. Greenewald, Jr. and T. J. Riley, dated April 1963
2. Brochure on Haynes Alloy No. 713c, published by Haynes Stellite Company, April 1959